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# Screen Cage Ion Plating (SCIP) and Scratch Testing of Polycrystalline Aluminum Oxide

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# SCREEN CAGE ION PLATING (SCIP) AND SCRATCH TESTING OF POLYCRYSTALLINE

## ALUMINUM OXIDE

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### SUMMARY

A screen cage ion plating (SCIP) technique was developed to apply silver films on electrically non-conducting aluminum oxide. It is shown that SCIP has remarkable throwing power; surfaces to be coated need not be in direct line-of-sight with the evaporation source. Scratch tests, employing a diamond stylus with a 200  $\mu\text{m}$  radius tip, were performed on uncoated and on silver-coated alumina. Subsequent surface analyses show that a significant amount of silver remains on the scratched surfaces, even in areas where high stylus loads produced severe crack patterns in the ceramic. Friction coefficients during the scratch tests were lower on the coated alumina indicating that this modification of the ion plating process should be useful for applying lubricating films of soft metals to electrical insulating materials. The very good throwing power of SCIP also strongly suggests general applicability of this process in other areas of technology, e.g., electronics, in addition to tribology.

### INTRODUCTION

High friction and wear are observed for most ceramics during unlubricated sliding. Therefore, a lubricant is required before they can be useful in tribological applications. One approach to reducing friction and wear has been to coat the ceramic with a soft metal film. For example, it has been reported by Erdemir et al. (ref. 2), that ion beam assisted deposition (IBAD) of silver provides an adherent film on alumina that is effective in mitigating sliding friction and wear. DellaCorte et al. (ref. 1) reported similar results with silver films on a thin interlayer of titanium, both applied to alumina by magnetron sputtering. IBAD and sputtering are essentially "line of sight" processes that only coat the surface in direct line of the ion source; they have very limited "throwing power." In this paper, we describe a study of silver films applied by ion plating, a process with very good throwing power.

Ion plating has been reported to produce adherent coatings on metals (refs. 4 and 6). However, ion plating is normally used to coat electrically conductive materials such as metals. Direct ion plating cannot be used to plate insulating ceramics. Therefore, a modified ion plating technique, which is capable of coating ceramics, was developed in the present study. This paper describes the modified technique that we call screen cage ion plating (SCIP). We also give preliminary evaluations of coating behavior under the very severe sliding conditions of scratch testing with a diamond stylus.

### MATERIALS AND PROCEDURES

The alumina is sintered, polycrystalline, commercial material. Its composition, by spectrographic analysis is 52.6 wt% Al, <0.05 ppm Fe, and <0.03 ppm Ti. This is about 99 wt%  $\text{Al}_2\text{O}_3$ ; x-ray diffraction revealed only  $\alpha\text{-Al}_2\text{O}_3$  to be present. The density is 3.78  $\text{g}/\text{cm}^3$  or about 95 percent of theoretical. The block was cut into 18 by 6 by 12 mm samples. No further surface grinding or polishing was used. The surface roughness ( $|R_a$ ) was 0.25 to 0.38  $\mu\text{m}$  and the measured Vickers hardness at 25 °C

and 100 g load was 1607 kg/mm<sup>2</sup>. Sasaki (ref. 5) reports a value of 1800 kg/mm<sup>2</sup>. This or greater variability is expected from various sources of alumina.

### Ion Plating of Silver

The effectiveness of the ion plating process stems from its ability to provide a high energy flux of ions and energetic neutrals which contribute to the adherence and desirable microstructure of the film. In the conventional ion plating process, the system consists of a dc-diode configuration. The specimen to be coated is an electrical conductor and is made the cathode of a high voltage dc-circuit. The evaporation source is the anode. Typical ion plating conditions are: 2 to 5 kV negative potential applied to the specimen; 0.2 to 0.5 mA/cm<sup>2</sup> current density and 10 to 20 mtorr argon pressure.

In this study, where silver had to be ion plated on a ceramic (insulator) substrate, the existing dc-diode system could not be used. Therefore, an indirect approach of employing a screen-cage technique was developed (fig. 1). The ceramic (Al<sub>2</sub>O<sub>3</sub>) substrate is mounted and surrounded with a 20 mesh silver screen cage that functions as an electronic grid. The negative potential is applied to this cage. A field-free region forms between the screen cage and the surface of the ceramic thus neutralizing the positive charge buildup on the ceramic surface by the emission of secondary electrons from the grid. The separation between the grid and the ceramic surface is critical, and generally, should be small on the order of 6 mm. If the spacing is large, a hollow cathode discharge is setup which generates excessive heating. The screen-cage ion plating (SCIP) conditions for depositing a silver film on the alumina substrate were the following: (-3000 V, 80 mA), effective cathode area approximately 48 cm<sup>2</sup>. Coating thicknesses in this program were 2 to 3 μm as measured on cross-section photomicrographs. Because of the high throwing power of the (SCIP) process, all exposed alumina surfaces were coated with silver. The cross section photomicrographs of figure 2 show that the surface 180° from the silver evaporation source was coated with about one-third the coating thickness on the side facing the source.

### Scratch Testing

Scratches were made on coated and uncoated surfaces. A commercial scratch tester was used. The scribe was a diamond stylus with a 200 μm tip radius and a 120° cone angle. Scratches were 10 mm long. One test mode chosen was that of a linear increase in load with distance at 100 N/min, and consequently a velocity of 10 mm/min. Scratches were also made at various constant loads in determining the effect of load on friction. All scratches were made at 25 °C in ambient air.

This tester was equipped with a tangential force measurement sensor. The tangential force was continuously recorded. The tangential force divided by the corresponding normal load is, by definition, the friction coefficient (μ) but the term scratch coefficient (μ\*) is sometimes considered to be more rigorously correct, especially for high loads where the energy dissipated by cracking and microfracture of the specimen is an added component of the tangential force. Either term will be used in this paper depending upon which is more appropriate in a particular context.

### Post-Scratch Examination

The scratches that were made at a 100 N/mm loading rate were examined by several standard methods. Photographs were made using the scanning electron microscope (SEM). The uncoated alumina



scratch was gold sputtered for examination. Scratches in the silver-coated samples were not gold sputtered.

Silver and alumina distribution in the bottom of the scratch were determined with an x-ray energy dispersive spectrometer (EDS) attached to the SEM. An area and line method was used. For the area method, the magnification of the SEM was adjusted (200X) so that only the bottom of the scratch was imaged at the lowest load (5 N) area analyzed. This magnification was maintained constant and EDS spectra were recorded at incremental distances along the length of the scratch. In this manner, relative intensities for silver and aluminum at progressively higher loads were determined. For comparison to the area method, continuous spectra were made along the length of the scratch center spectra recorded along this line. In both cases, the spectra were reduced to relative silver and aluminum contents by software. The silver and aluminum content was normalized to 100. A spot mode was also used to determine the element content of chosen features.

## RESULTS AND DISCUSSION

### Friction During Continuously Variable Load Tests

Examples of scratch test friction data for bare and silver coated alumina are shown in figure 3. The friction coefficient decreases with load up to 20 or 30 N, then levels out at about 0.15 for bare alumina and about 0.10 for silver-coated alumina. The negative slope at lighter loads agrees with elasticity theory which states that, in the elastic contact of a ball on a flat, the contact area,  $A$ , increases only as the two-thirds power of the load,  $W$ . Since the friction force,  $F_T$ , is equal to the product of a constant shear strength and the area of contact, the friction force also increases as the two-thirds power of load or:  $F_T \propto W^{2/3}$ . Therefore, the friction coefficient ( $F_T/W$ ) varies as  $W^{-1/3}$ . This proportionality was used to calculate curve D on figure 3 where  $\mu$  was normalized to 0.16 at 10 N and the remaining values were computed using the  $\mu \propto W^{-1/3}$  relationship. The calculated curve D superimposed the data curve A for silver-coated alumina from 10 to 30 N. At higher loads, the curves diverge indicating that elastic behavior no longer predominates. The plateau and eventual increase in friction coefficient is probably attributable to substantial cracking and microfracture of alumina at the higher loads. A similar comparison for curve B (normalizing  $\mu$  to 0.14 at 5 N) shows a divergence from elastic behavior above a 20 N load.

Curve B is for alumina coated with silver over only part of the scratch length, and shows a very sharp rise in friction coefficient as the stylus traverses from the coated to the uncoated surface area.

In nine scratch tests each on bare and coated alumina the scatter in friction coefficients in the mid-load plateau was 0.12 to 0.15 for bare alumina and 0.05 to 0.10 for silver-coated alumina. Seven out of the nine measurements for coated alumina were skewed to 0.09 to 0.10.

### Friction at Constant Loads

In order to check the validity of scratch coefficients (determined from variable load experiment) as a measure of the friction, a series of scratches were made at constant loads. Separate scratches were made in normal load increments of approximately 2 N to a maximum of 20 N. The results for uncoated and I.P. silver-coated alumina are shown in figure 4. After the initial negative slope at lighter loads, the friction coefficients plateaued at  $0.15 \pm 0.01$  for uncoated alumina and  $0.10 \pm 0.03$  for coated alumina in

good agreement with the scratch coefficients obtained over the same load range in the continuously variable load experiments.

### Photomicroscopy and Surface Analysis

The thicknesses of the ion-plated silver as measured on cross section photomicrographics was about 3  $\mu\text{m}$ . A composite SEM photomicrograph of scratch segments on the silver-coated surface is presented in figure 5. Examination of the areas outside the scratch reveals the silver deposit is a noncontinuous film with many small voids. In the scratch it is clear that plowing of the silver had occurred. Spot EDS analysis reveals the white island-like features at the bottom of the scratch are silver and the black areas are the aluminum oxide substrate. Even in the 98 to 100 N load area, silver still remains in the bottom of the scratch as islands even though most of the silver has been plowed away. This conclusion is reinforced by the EDS data presented in figure 6. The silver in the bottom of the scratch decreases only slightly with increasing load and even at 100 N the scratch has retained silver. The area and line methods of analysis are in good agreement.

SEM photomicrographs of a scratch made on uncoated alumina are presented in figure 7. Figure 7(a) shows the texture of the surface outside the scratch and is that resulting from preparation. The small flat zones are cut grains. Photomicrographs in the scratch on bare alumina are presented in figures 7(b) and (c) at a load near 100 N. Many large flat islands are present. These formations could be the result of plastic deformation of the alumina (ref. 3) or compaction of microfractured particles (ref. 2). The second explanation is reinforced by the presence of many very fine particles trapped between the islands. When silver is present, the compacted islands are coated with a thin layer of silver which provides lubrication. Cracks were not easily observed on the surface, but extensive crack patterns and subsurface damage were observed in cross sections of the scratched specimens.

Cross sections of scratches made under 5 and 80 N loads on silver-coated alumina are shown in figure 8. At 5 N, no cracks occurred; at 80 N, an extensive crack pattern was generated. (A study to determine the critical load and surface tensile stress to initiate and propagate cracks in uncoated and silver-coated alumina is described in a companion paper by Sliney and Spalvins (ref. 7). Surface profilometry traces across scratches made at various loads on silver-coated alumina are shown in figure 9. The scratch depths for 50 and 90 N loads are approximately equal to the thickness of the silver and are about 3  $\mu\text{m}$  deep. Little or no alumina was worn away, but much subsurface damage is evident. In spite of this damage, as previously shown, the surface of the scratch retains a significant amount of silver over the entire scratch length. A surface profile of a scratch made under a 60 N load on uncoated alumina shows removal of uncoated alumina to a depth of 2  $\mu\text{m}$  (fig. 10).

The microfracture damage in silver-coated alumina is less severe than in uncoated alumina, especially at loads up to about 30 N. This is attributed to lower friction on the coated surfaces and consequently lower surface tensile stress during sliding. It is expected, therefore, that ion plated silver and other soft metal coatings will be even more beneficial to ceramic on ceramic sliding combinations that typically have much higher friction ( $\mu = 0.5$  to 1.0) than diamond on alumina.

### SUMMARY OF RESULTS

A screen cage ion plating (SCIP) technique was developed and successfully used to deposit films of silver on electrically nonconducting aluminum oxide (alumina). In this technique, the ceramic or other nonconductor to be coated is enclosed in a cathodic cage made of silver screen. Positive silver ions are



accelerated to the cage where many of them pass through the openings in the screen grid and deposit on the ceramic. Surface analyses after scratch tests of the coated specimens show retention of silver on the scratches even where extensive microfracture and cracking of the alumina substrate occurred. Friction coefficients during scratch tests were lower on coated than on uncoated alumina indicating that screen cage ion plating is a useful surface modification technique for lowering the friction and mitigating tensile stress-induced microfracture wear.

### CONCLUDING REMARKS

1. A modification of the ion plating process was developed that enables ion plating of metals on electrically nonconductive ceramics. We call this technique "screen cage ion plating (SCIP)."

2. Silver films were deposited on aluminum oxide by this technique. An advantage of the SCIP method over line-of-sight processes such as DC sputtering is its excellent throwing power. Surfaces 180° away from the evaporation source were found to be coated with as much as 30 percent of the coating thickness on surfaces facing the evaporation source.

3. During scratch tests, the friction coefficient of a diamond stylus against alumina was reduced by 30 to 50 percent by lubricating films of silver deposited by the SCIP process. Reducing the friction mitigates the surface tensile stresses which are responsible for microfracture wear of brittle materials.

### ACKNOWLEDGMENT

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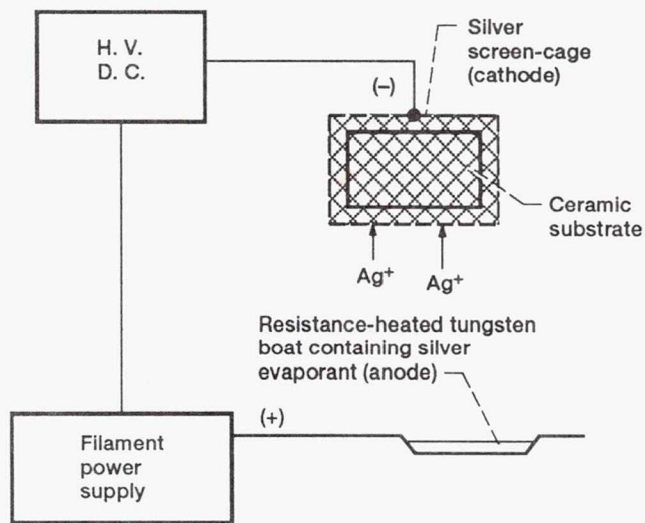


Figure 1.—Schematic for screen-cage ion plating of ceramics.

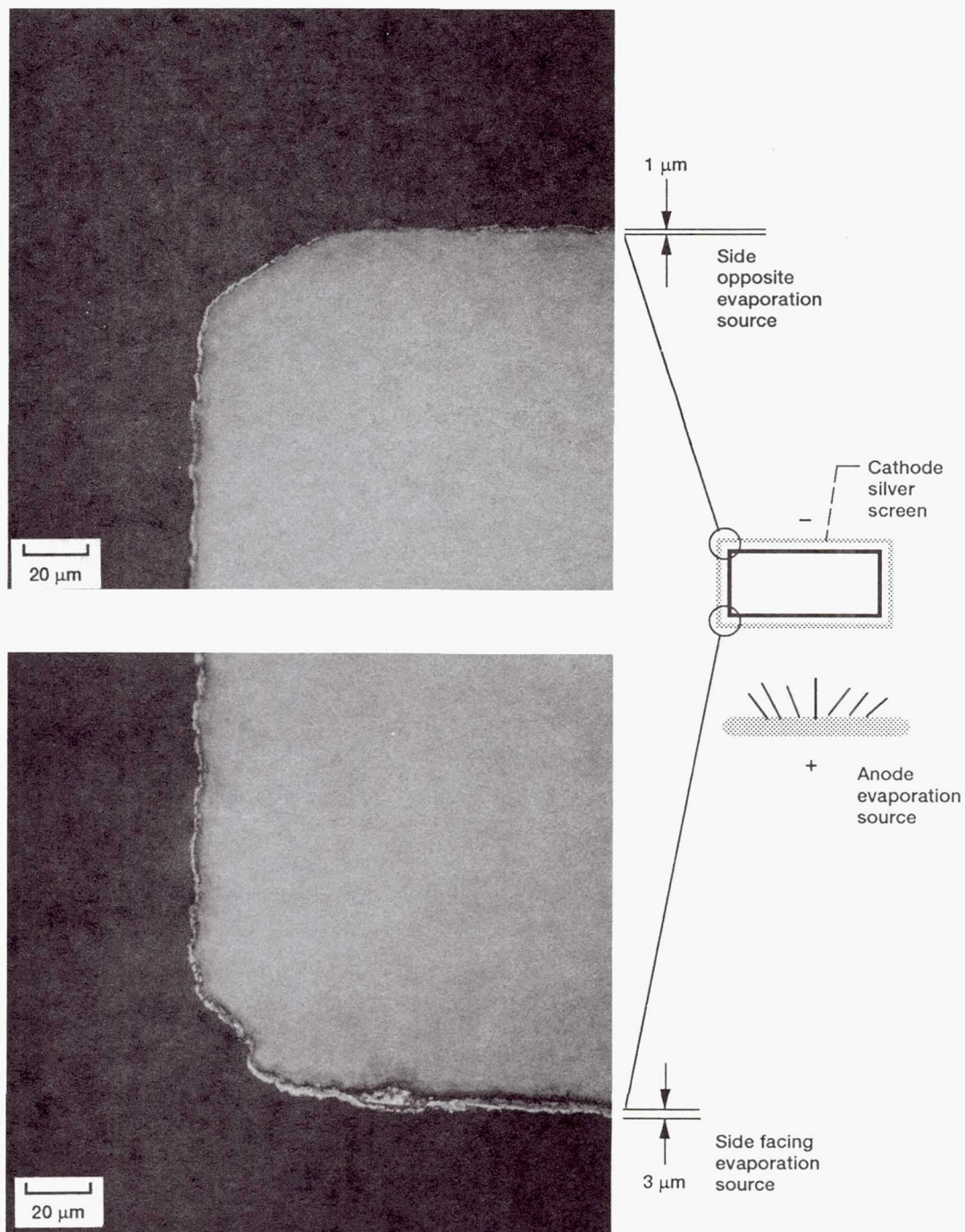


Figure 2.—Illustration of excellent "throwing power" (complete substrate coverage) of the "SCIP" process.



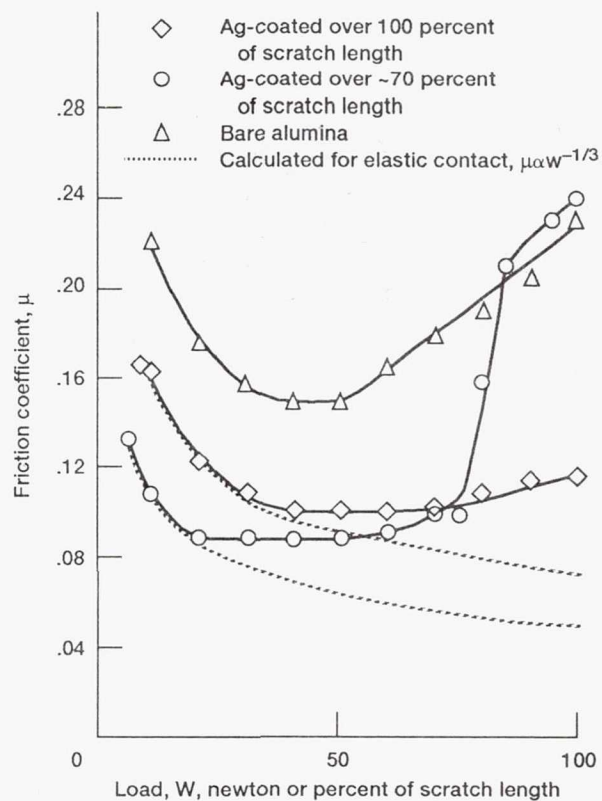


Figure 3.—Continuously-variable-load scratch test of bare and silver coated polycrystalline aluminum oxide; 25 °C, 0.2 mm radius tip on stylus, 100 N/min load rate, 10 mm/min velocity.

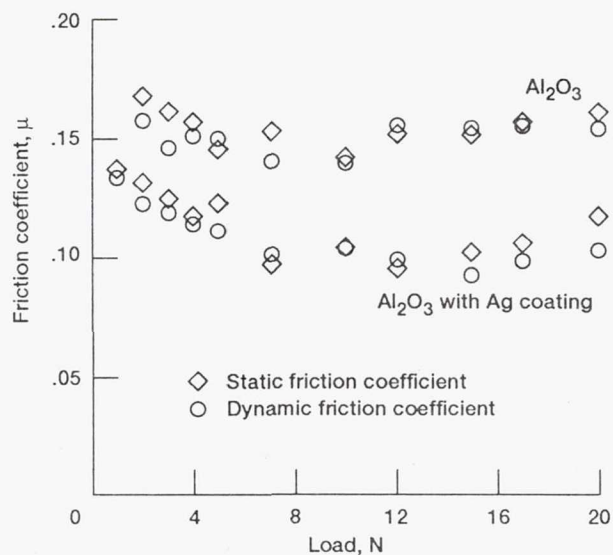


Figure 4.—Constant load scratch tests of bare and silver coated aluminum oxide; 25 °C, 0.2 mm radius tip on diamond stylus, 12 mm/min velocity.

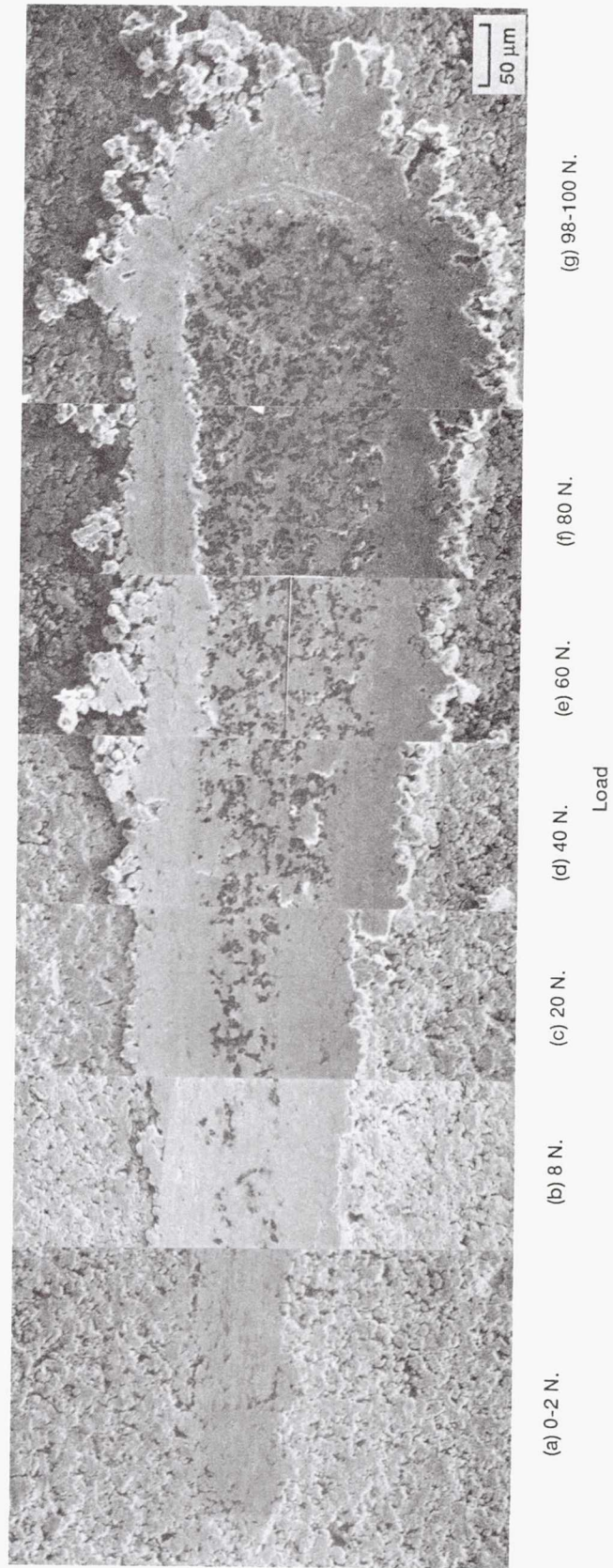


Figure 5.—SEM photographs of scratch path segments on silver-coated polycrystalline aluminum oxide.



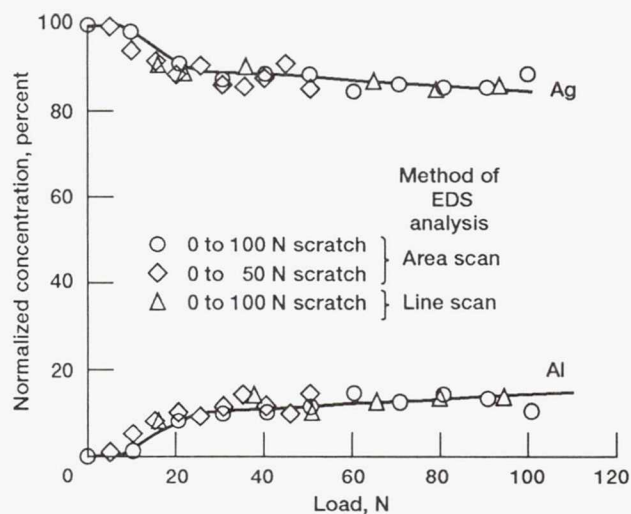


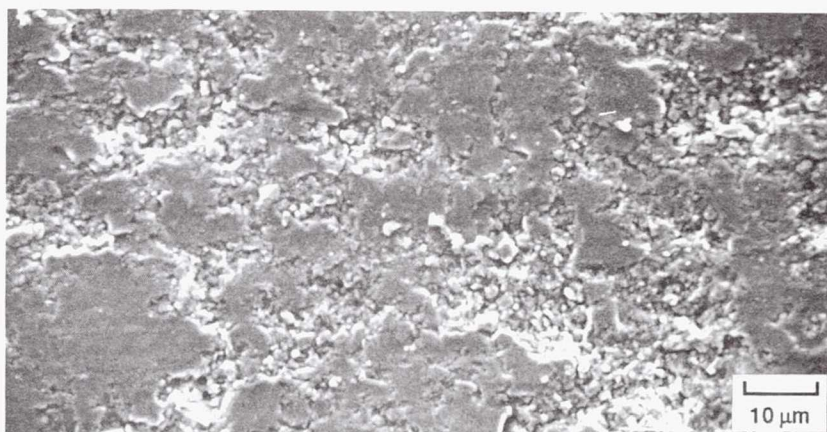
Figure 6.—Ag and Al in center of scratch for Ag coated polycrystalline  $\text{Al}_2\text{O}_3$  as determined by EDS area and line scan analysis. (Silver-aluminum normalized to 100)



(a) Area outside scratch.



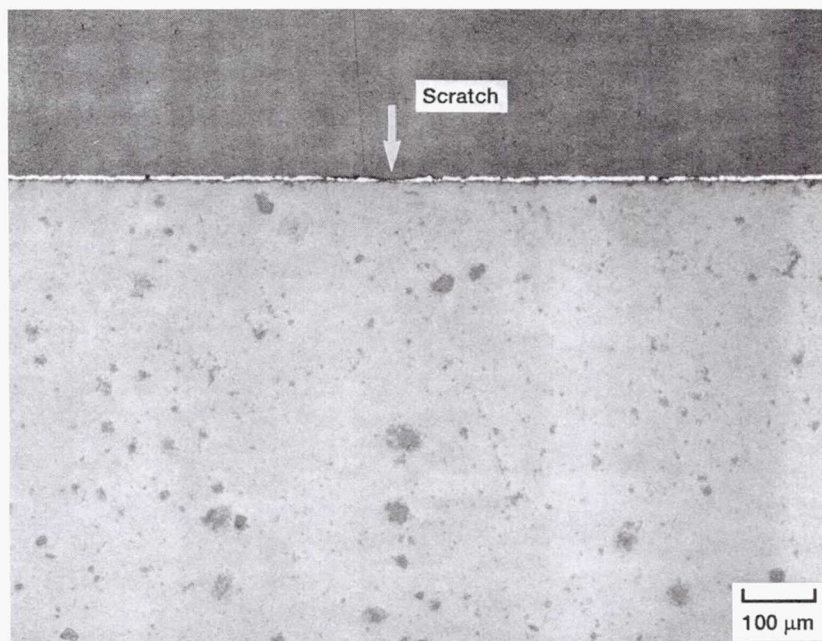
(b) Scratch near 100 N load.



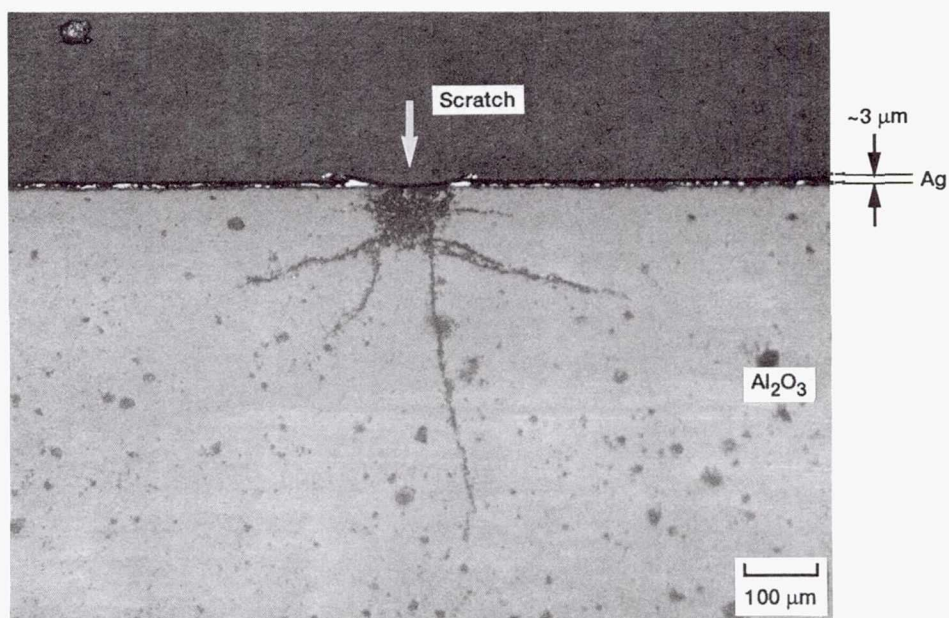
(c) Bottom of scratch near 100 N load.

Figure 7.—SEM photographs of scratch on the uncoated alumina. (Au coated for photographs)





(a) 5 N load.



(b) 80 N load.

Figure 8.—Cross sections of scratches on silver-coated alumina polished, unetched.

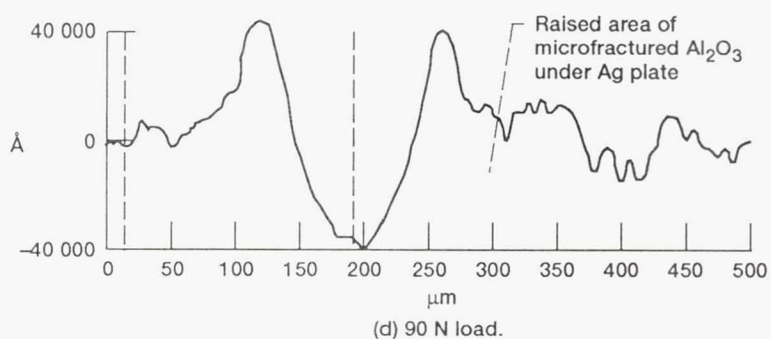
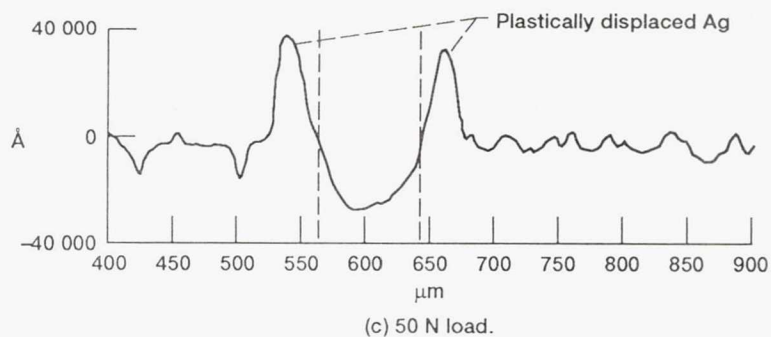
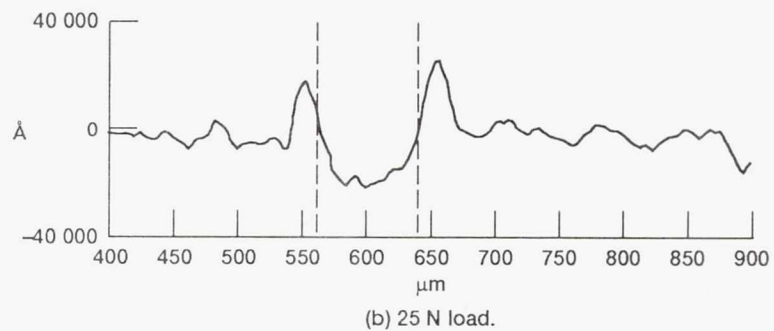
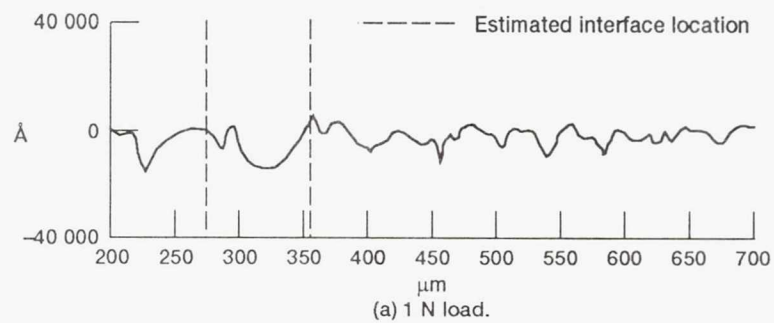


Figure 9.—Profilometer traces across scratches made at various loads by a 0.2 mm radius diamond on silver-ion-plated alumina.



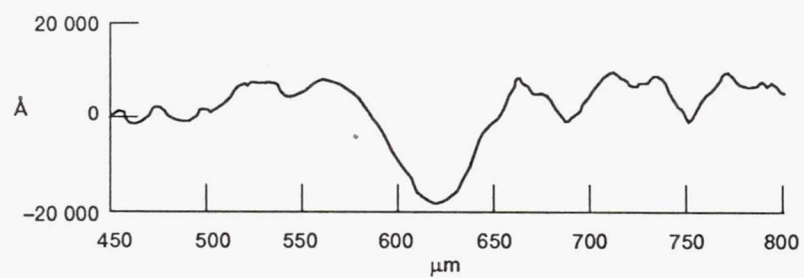


Figure 10.—Profilometer trace across scratch made at 60 N load by a 0.2 mm radius diamond on uncoated alumina.

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